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AIRBORNE RESPIRABLE COMBUSTIBLE DUST MASS DETERMINATION - CHARACTERIZATION OF ASHING PROPERTIES OF SILVER MEMBRANE AND ACRYLIC BOUND GLASS FIBER FILTERS

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ABSTRACT

The determination of the combustible component of airborne respirable dust is an important issue. Ashing of airborne dust samples at a high temperature is one way of obtaining the respirable combustible dust (RCD) fraction. The work presented here gives the results of preliminary laboratory testing of silver membrane and one type of glass fiber filter submitted to ashing tests. Silver membrane filters are presently used in many Canadian mines for sampling airborne respirable silica dust. Routine ashing analyses performed on these same filters after they have been analyzed non-destructively for silica could yield worker's RCD exposure. The Elliot Lake Laboratory also uses glass fiber substrates containing an acrylic binder in cascade impactors for mine dust size distribution characterization. In order to be able to determine mine RCD size distribution the ashing properties of this type of glass fiber filter were also examined. Blank silver membrane filters have shown mass losses of the order of 0.1% when ashed for four hours at 500°C. The glass fiber filters tested showed significant mass losses after the first ashing session probably because of ashing of the acrylic binder. Subsequent sessions, however, demonstrated that losses of the order of 0.1% could be expected. An ashing protocol is given which suggests selecting field blanks from batches of silver membrane and glass fiber filters to be used in the field. Glass fiber filters should be pre-ashed in order to avoid complications arising from the loss of mass associated with the acrylic binder.

Key words: Respirable dust.

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DETERMINATION DE LA MASSE DES POUSSIERES COMBUSTIBLES RESPIRABLES ATMOSPHERIQUES - CARACTERISATION DES PROPRIETES DE REDUCTION EN CENDRES DES FILTRES A MEMBRANE EN ARGENT ET DES FILTRES A FIBRES DE VERRE LIEES A L'ACRYLIQUE

M. Grenier*

RESUME

La détermination de la composante combustible des poussières respirables atmosphériques est une question importante. La réduction en cendres d'échantillons de poussières atmosphériques à une température élevée est un moyen d'obtenir la fraction de poussières combustibles respirables (PCR). Le travail présenté ici décrit les résultats d'essais de laboratoire préliminaires sur la réduction en cendres d'une membrane en argent et d'un type de filtre en fibre de verre. De nombreuses mines canadiennes sont actuellement munies de filtres à membrane en argent pour échantillonner la poussière de silice respirable dans l'air. Les analyses courantes de réduction en cendres effectuées sur ces mêmes filtres après dosage non destructif de la silice pourraient indiquer le niveau d'exposition des travailleurs aux PCR. Le Laboratoire d'Elliot Lake utilise aussi des substrats en fibre de verre contenant de l'acrylique comme liant dans des impacteurs à cascade pour caractériser la granulométrie des poussière de mine. Pour pouvoir déterminer la granulométrie des PCR, il a fallu aussi examiner les propriétés de réduction en cendres de ce type de filtre en fibre de verre. Des filtres à membrane d'argent vierges ont été réduites en cendres pendant quatre heures à 500 °C, et les pertes de masse étaient de l'ordre de 0,1 %. Les filtres en fibre de verre examinées ont présenté des pertes de masse importantes après la première séance de réduction en cendres, probablement à cause de la réduction en cendres du liant en acrylique. Les séances ultérieures ont toutefois révélé qu'il fallait s'attendre à des pertes de masse de l'ordre de 0,1 %. Le protocole de réduction en cendres présenté propose de choisir sur le terrain des filtres vierges à mêmes des lots de filtres à membrane en argent et de filtres en fibre de verre destinés à une utilisation sur le terrain. Les filtres en fibre de verre devraient être réduits en cendres au préalable pour éviter les complications découlant de la perte de masse associée au liant en acrylique.

Mots clés: poussières respirables

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INTRODUCTION

Determination of the combustible components of airborne respirable dust is fast becoming an important issue in mining enviroment safety and health as increasing attention is being focused on the liquid and solid products of diesel engine exhaust. In recent years, the health implications of exposure to diesel exhaust contaminants have resulted in mining standards being prescribed for ventilation requirements in diesel areas of underground operations and the maximum concentration of contaminants allowed in raw exhaust. More recently, the Air Quality Index (AQI) was developed as a measure of the overall effect of major contaminants from diesel exhaust (particulate and gaseous) on mine air quality. (1).

Actual field measurements show that Respirable Combustible Dust (RCD) from diesel soot may contribute as much as 57% to the AQI value. Because of these findings and a greater sense of awareness in safety and health issues it is expected that new regulations are forthcoming to monitor the exposure of workers to soot and other RCDs.

In order to be able to develop these guidelines and apply them successfully, more work needs to be done on the development of methods for measuring the combustible fraction of airborne dust. Also, a better knowledge of the chemical and physical characteristics of diesel soot will help in the design and development of better sampling and monitoring equipment for this contaminant.

In order to address these two points, this report gives laboratory results of ashing procedures applied to sampling filters and substrates. These preliminary results are used here to help develop a sampling protocol for the determination of RCD concentration in mine air as well as RCD size distribution. Although these results could potentially help in the eventual implementation of routine sampling schedules for mine worker exposure monitoring, they were developed mainly as a research and development aid.

GENERAL ASHING PROCEDURE

In determining the mass of RCD on sampling media, several factors need to be taken into account, for example, the effect of atmospheric moisture on filter mass, the time and temperature required for complete ashing of RCD's. Other technical details such as oven type, size and integrity may also influence the results of ashing.

In theory, a filter or sampling media that is sturdy, not affected by variation in relative humidity and free from components that may be removed by exposure to high temperatures would be an ideal candidate for such a task as RCD determination. All that would be required is pre-weighing of the media, field sampling, weighing to establish the mass of total respirable dust, ashing for a few hours at a temperature of 500°C and post-weighing to determine the mass of RCD.

In practice, however, this perfect filter material is not available and provision must be made in order to accurately measure the RCD component of mine dust. Amongst these a judicious choice of filter material is important. The filter must be sturdy to be able to withstand normal manipulation. Secondly, the effect of relative humidity on filter mass must be known, and finally consideration must be given to pre-conditioning techniques such as pre-ashing in order to remove combustible components of the filter media prior to sampling. The effect of pre-ashing on filter material properties should also be kept in mind.

RCD CONCENTRATION MEASUREMENT

Respirable silica exposure regulations have been in effect for some time. In Ontario, for example, respirable dust samples are collected at a flow rate of 1.7 L/min on 5 μm silver membrane filters. A 10 mm nylon cyclone is used to remove the non-respirable fraction of airborne dust. Because of their sturdy nature and the fact that relative humidity does not significantly

affect their mass (3), silver membrane filters could potentially be used for the assessment of RCD exposure of miners. With more attention being given to weighing of samples, routine monitoring of workers for exposure to RCDs could be implemented as an extension of the silica monitoring program.

SIZE DISTRIBUTION MEASUREMENT

Gravimetric assessment of airborne dust size distribution may be performed by using so-called cascade impactors. These instruments separate airborne dust into several size ranges by having dust laden air pass through a stack made up of several stages. Each stage consists of a plate with slots or circular openings which allow the air to circulate through the instrument. Progressively smaller openings in subsequent stages accelerate the airflow. Following each stage is a collection substrate which forms an impaction surface. Depending on air velocity through the jet or opening and particle mass, individual particles will follow the airstream into the next stage or will impact on the collection substrate. Presently the Elliot Lake Laboratory uses the Sierra series 210, 10-stage cascade impactor and the Marple personal cascade impactor.

The choice of collection media or impaction substrate is dictated by the application and what type of dust is being sampled. Several materials are available, such as PVC, mylar, glass fiber and even stainless steel. For the purpose of ashing, glass fiber filter material is probably the best candidate.

Several types of glass fiber materials are available and pros and cons have been examined in choosing the most adequate candidate. Binder free glass fiber filters are quoted as having high operating temperature characteristics (4) which would seem to be an asset when dealing with ashing. Years of experience with cascade impactors have demonstrated, however, that considerable mass loss may occur during normal manipulation and sampling procedures.

A sturdier glass fiber filter was chosen which could stand up to the

requirements of cascade impactor sampling. This filter consists of borosilicate glass fibers held by an acrylic binder. These filters are quoted as having an operating temperature of 135°C and major mass losses will occur upon ashing as the binder is burned from the filter matrix. Pre-conditioning of these filter discs by ashing them prior to sampling has to be examined and the effect on material strength established. Although glass fiber filters have been shown to have a stable mass at relative humidities lower than 50% (5), it is suspected that considerable mass increases may result from exposure to higher relative humidity environments. For this reason, weighing during these tests was conducted at an ambient relative humidity of around 30%, and filters were left to condition and stabilize for a period of several hours prior to weighing.

EXPERIMENTAL PROCEDURE AND RESULTS

Two types of filter materials were evaluated during the ashing test. Silver membrane filter discs of various diameter and pore sizes, and Gelman Metrigard glass fiber collection substrates were used throughout the investigation. It should be mentioned at this point that actual field tests were not performed and tests were conducted strictly on unused material.

Three types of silver membrane filters were chosen. Batch 1 consisted of 9, 13 mm, 5 μm pore size filters. Similarly, 9, 25 mm, 1.2 μm filter discs were used to make up Batch 2. Batch 3 consisted of 9, 25 mm, 5 μm filter discs. Whereas Batch 3 filters were fresh out of a sealed package, Batches 1 and 2 were selected from old stock that had been left exposed to the air for years. As a result of this, several of the old filters were severely affected by silver sulphide deposits. The extent of the problem varied from a slight darkening of the filter edges to severe total darkening of one side of the filter surface. The old stock was purposely selected to assess the effect of ashing on silver salts formed through time on filter sufaces. Ashing of

affected filters always resulted in the dark stains being removed and the extent of mass change due to this phenomenon had to be assessed. Silver membrane filters used during this are described in more detail in Table 1.

Silver membrane filters were selected, left to condition in the weighing room for a period of several hours, and pre-weighed on a Mettler AE163 balance with manufacturer quoted readability and a reproducibility rating of 0.01 mg and 0.02 mg, respect ely. Filters were then ashed for a four hour period at a temperature of 500°C in a Thermolyne 2000 oven. Filters were conditioned overnight and re-weighed. The mass difference was noted and 2 more 4 hour ashing period were performed on the same filter batches, with weighing being performed before and after ashing. The results of silver membrane filter ashing are shown in Table 2.

Glass fiber cascade impactor substrates were cut and left to condition for a period of 24 hours. Four batches were prepared, each of which consisted of 8 slotted 47 mm discs and 1 unslotted 47 mm back filter. In actual field use one impactor run would require 8 to 10 slotted substrates and one back filter before air leaves the impactor. The four slots on slotted substrates were disposed radially in a cross pattern and each measured 3 mm in width and 10 mm in length. The first two batches were treated similarly to the silver membrane filters and were ashed twice for 2 hour periods.

Batches 3 and 4 were exposed to three 4 hour ashing sessions. When glass fiber filters were being tested records of relative humidity and temperatures were kept. The results of glass fiber filter ashing for Batches 1 and 2 are given in Table 3, and those for Batches 3 and 4 are given in Table 4.

DISCUSSION

The observations and results pertaining to silver membrane filters are given in Tables 1 and 2. From these Tables it is apparent that the age,

diameter and pore size of silver membrane filters does not noticeably affect the mass lost during the first ashing session.

This loss of mass seems to be a more or less linear function of the original filter mass and the average mass loss has a degree of variability of the order of magnitude of the balance's reproducibility. It seems, therefore, that the amount of filter material lost upon ashing of actual field samples could be accurately determined by ashing field blanks from the same batch. New 25 mm, 5 μm silver membrane filters lost, on average, 0.15 mg $\pm$0.02 mg. This mass loss represents, on average, 0.1% of the original filter mass.

Results of Metrigard glass fiber filter ashing are given Tables 3 and 4. From Table 3 it became apparent that a 2 hour ashing session was perhaps not long enough to accurately remove combustible components in the filter matrix. Part of the reason for the lack of agreement between the results of Batches 1 and 2 is due to the fact that the tray holding Batch 1 was positioned too close to the oven door where the oven temperature may have been lower. In any case, results from Batches 1 and 2 indicate a large mass loss during the first ashing session and a variability that is far too high to be acceptable.

Table 4 lists the results of three 4 hour ashing sessions performed on glass fiber filter batches 3 and 4. Again, the first ashing session shows large mass losses of the order of 6.5% of the original filter mass. The real problem with the first ashing session is the variability of $\pm$0.33 mg. This value is of the order of magnitude of what would be expected to be collected on impactor stages in mine return air and areas where diesel equipment is in use. It is, therefore, not conceivable to presume that such collection substrates could be used to measure RCDs without pre-ashing.

The second ashing session yielded results which were extremely reproducible when comparing both batches. In this instance, mass losses of 0.13 mg $\pm$0.02 mg were measured. These results are in fact very similar to

those measured after the first ashing session with silver membrane filters. It is expected, therefore, that the glass fiber filters with acrylic binder may be used in cascade impactors to determine RCD size distribution.

CONCLUSIONS

The results of ashing performed on blank silver membrane and acrylic bound glass fiber filters are encouraging. It would now seem that the best procedure to follow for RCD sampling using silver membrane filters is as follows:

1. Select 5 to 10 filters from the batch to be used in the actual field survey.
2. Weigh all filters under controlled conditions of relative humidity and temperature, allowing the filters to condition for a few hours.
3. Perform field sampling.
4. Condition field samples and weigh to determine total respirable dust concentration.
5. Conduct X-ray diffraction analyses to determine the concentration of respirable quartz.
6. Re-weigh to ensure no mass was lost during x-ray manipulation.
7. Ash field samples and field blanks.
8. Weigh samples and blanks.
9. Use blank mass loss to correct field filter mass loss and determine the amount of RCD on the filter.

The method to use for the determination of RCD size distribution is similar except that field blanks and field sampling filters made from glass fiber material should be pre-ashed and there is no need in this case to worry about X-ray diffraction analysis.

For the glass fiber filter material tested here the variability measured on the second ashing session was 0.02 mg. Depending on the cascade

impactor's flow rate and the amount of small RCD particles in the air, this value could be as much as 20% of the RCD collected on lower stages. It should be kept in mind, however, that 0.02 mg is also the manufacturer quoted balance reproducibility (standard deviation) which means that at this point the balance is as much a source of uncertainty as the ashing procedure.

Visual inspection and manipulation of ashed acrylic bound glass fiber filters revealed that filter strength and integrity did not seem to be affected and that impaction substrates made from this ashed material should stand up to field use. It should be noted also that weighing of glass fiber material should be performed in a small room where the humidity can be kept below 30-35% by using a dehumidifier. This would seem to be a better alternative to drying filters using a desiccation enclosure, since rapid mass increase will probably occur as soon as the filters are removed from the desiccator to be weighed.

Field trials scheduled in the near future will help determine if the methods presented here will allow RCD concentration and size distribution to be measured with an acceptable degree of accuracy. It is expected, however, that field trials will probably allow for improvements to be made on the suggested methods.

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Table 1 - Qualitative estimate of the surface area of silver membrane filters affected by silver salt deposits.

Batch/Filter	Diameter (mm)	Pore(μm)	Silver Salts (0-10)*
1-1	13	5.0	2
1-2	13	5.0	2
1-3	13	5.0	2
1-4	13	5.0	5
1-5	13	5.0	10
1-6	13	5.0	7
1-7	13	5.0	9
1-8	13	5.0	2
1-9	13	5.0	2
2-1	25	1.2	2
2-2	25	1.2	2
2-3	25	1.2	2
2-4	25	1.2	2
2-5	25	1.2	2
2-6	25	1.2	2
2-7	25	1.2	2
2-8	25	1.2	2
2-9	25	1.2	2
3-1	25	5.0	0
3-2	25	5.0	0
3-3	25	5.0	0
3-4	25	5.0	0
3-5	25	5.0	0
3-6	25	5.0	0
3-7	25	5.0	0
3-8	25	5.0	0
3-9	25	5.0	0

*Percentage of surface affected by silver salt deposits estimated visually and ranked between 0 and 10, 0 being 0% of surface and 10 being 100% of surface affected by staining.

Table 2 - Results of silver membrane filter ashing procedures

Batch/ Filter	Mass (mg)	Δ Mass (mg) from Ashing Session No. 1	No. 2	No. 3	Δ Mass Total (mg)
1-1	47.52	0.08	0.02	0.05	0.15
1-2	50.19	0.09	0.01	0.03	0.13
1-3	49.01	0.04	-0.01*	0.05	0.08
1-4	47.81	0.07	0.00	0.04	0.11
1-5	40.97	0.03	0.02	0.05	0.10
1-6	49.01	0.04	0.06	0.06	0.16
1-7	50.83	0.04	0.00	0.04	0.08
1-8	49.39	0.04	0.01	0.04	0.09
1-9	43.75	0.03	-0.01	0.05	0.07
Ave ± S.D.**		0.05±0.02	0.01±0.02	0.04±0.01	0.11±0.03
2-1	105.59	0.14	0.08	0.13	0.34
2-2	106.18	0.12	0.07	0.07	0.27
2-3	109.91	0.09	0.06	0.11	0.27
2-4	116.31	0.03	0.04	0.08	0.15
2-5	97.36	0.07	0.06	0.10	0.22
2-6	109.25	0.07	0.06	0.10	0.23
2-7	113.38	0.06	0.04	0.12	0.22
2-8	105.36	0.07	0.05	0.12	0.24
2-9	108.87	0.10	0.07	0.14	0.31
		0.08±0.03	0.06±0.01	0.11±0.02	0.25±0.06
3-1	121.78	0.17	0.10	0.02	0.30
3-2	118.62	0.16	0.04	0.02	0.23
3-3	107.39	0.12	0.09	0.03	0.23
3-4	112.22	0.14	0.09	0.02	0.26
3-5	112.12	0.13	0.09	0.05	0.27
3-6	108.02	0.15	0.08	0.02	0.25
3-7	111.66	0.15	0.12	0.01	0.29
3-8	103.78	0.16	0.12	0.01	0.29
3-9	103.61	0.19	0.12	0.02	0.32
		0.15±0.02	0.09±0.02	0.02±0.01	0.27±0.03

* Negative Δ Mass indicates net mass gain.
** Average and standard deviation.

Table 3 - Results of glass fiber collection substrate ashing procedure for batches 1 and 2 (two 2 hour ashing sessions).

Batch/ Filter	Mass (mg)	Δ Mass (mg) from Ashing Session No. 1	No. 2	Δ Mass Total (mg)
1-1	121.24	7.59	0.10*	7.68
1-2	119.17	7.43	0.13	7.56
1-3	121.96	7.97	0.11	8.08
1-4	119.08	7.25	0.09	7.34
1-5	115.97	7.35	0.11	7.46
1-6	118.30	7.79	0.14	7.93
1-7	113.54	7.46	0.16	7.62
1-8	113.33	7.50	0.16	7.66
1-B**	119.58	8.20	0.22	8.42
Average ±S.D.		7.61±0.31	0.14±0.04	7.75±0.34
2-1	118.96	7.66	-0.01	7.65
2-2	121.52	7.69	0.01	7.68
2-3	121.72	7.68	0.01	7.67
2-4	110.37	7.51	0.01	7.50
2-5	121.76	7.53	0.01	7.52
2-6	110.62	7.60	0.00	7.60
2-7	117.98	8.02	0.00	8.02
2-8	120.31	7.71	-0.02	7.73
2-B	119.67	8.37	0.00	8.37
Average ±S.D.		7.75±0.27	-	-

Relative humidity 26%, temperature 24°C.

*Batch 1 results show a large Δ Mass during the second ashing when compared with Batch 2 results. This was caused by Batch 1 filters being positioned too close to the front of the oven during the first ashing session, resulting in incomplete combustion of some of the combustible components of the filter matrix, and was evident from brownish deposits on the filters.

**Denotes back filter.

Table 4 - Results of glass fiber collection substrates ashing procedure for batches 3 and 4 (three 4 hour ashing sessions).

Batch/ Filter	Mass (mg)	Δ Mass (mg) from Ashing Session No. 1	No. 2	No. 3	Δ Mass Total (mg)
3-1	120.54	7.67	0.11	0.03	7.81
3-2	120.51	7.76	0.13	0.05	7.94
3-3	113.23	7.58	0.16	0.07	7.81
3-4	119.32	7.41	0.14	0.04	7.59
3-5	112.69	7.62	0.16	0.04	7.82
3-6	118.54	7.91	0.13	0.06	8.10
3-7	110.60	7.46	0.09	0.07	7.62
3-8	117.92	7.54	0.14	0.02	7.70
3-B	121.13	8.71	0.13	0.03	8.87
Average ±S.D.		7.74±0.39	0.13±0.02	0.04±0.02	7.92±0.39
4-1	118.13	7.98	0.12	0.04	8.14
4-2	110.67	7.56	0.14	0.02	7.72
4-3	118.56	7.60	0.13	0.07	7.80
4-4	119.48	7.69	0.16	0.05	7.90
4-5	119.07	7.54	0.17	0.04	7.75
4-6	119.89	7.54	0.14	0.04	7.72
4-7	117.33	8.04	0.15	0.04	8.23
4-8	118.13	8.06	0.16	0.02	8.24
4-B	119.83	8.24	0.13	0.02	8.39
Average ±S.D.		7.80±0.27	0.14±0.02	0.04±0.02	7.99±0.26

Relative humidity 28%, temperature 24°C.